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A NICKEL AND SILICON RICH PHASE IN IRRADIATED FV548 STEEL, (U)
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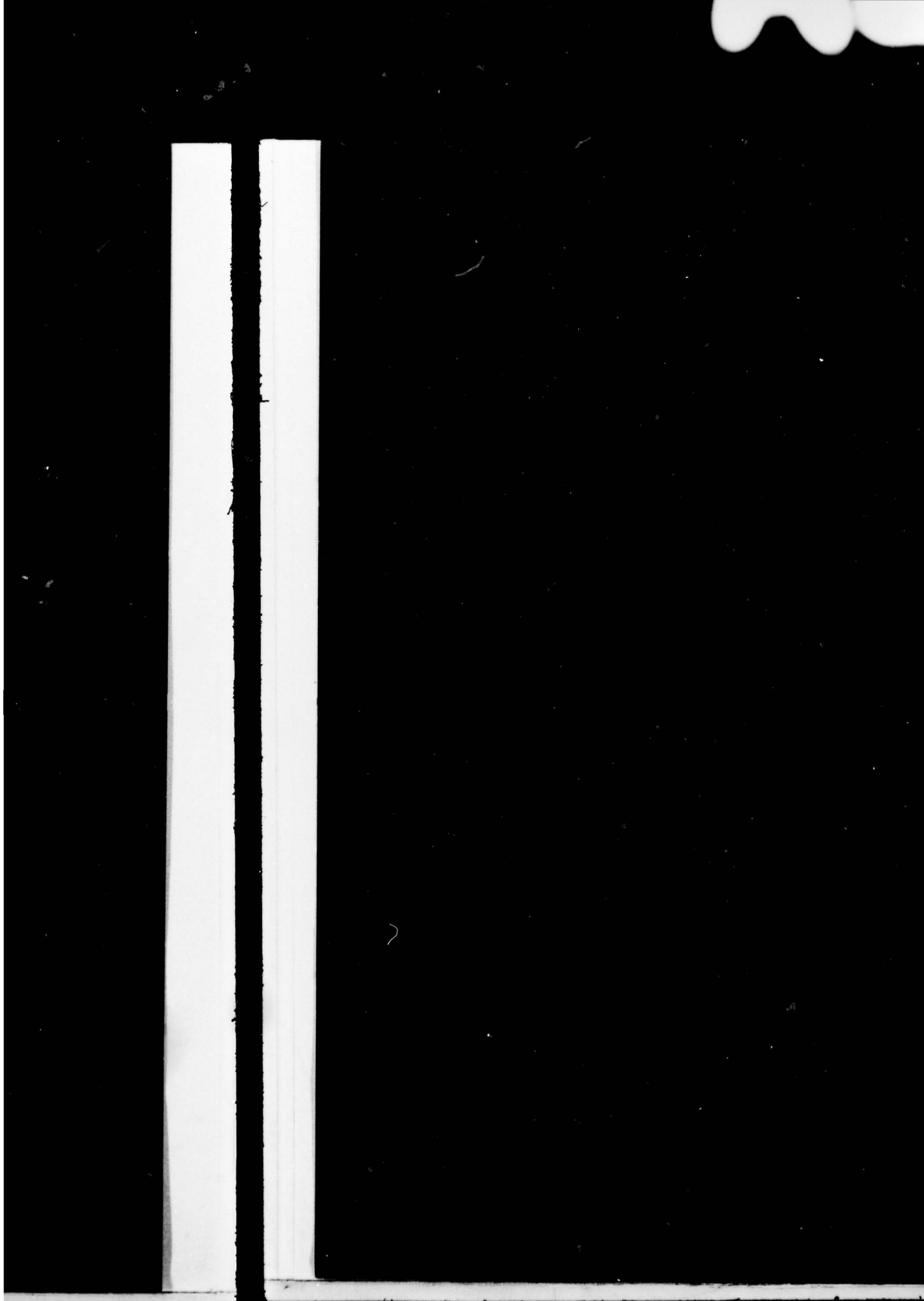
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6 A NICKEL AND SILICON RICH PHASE IN IRRADIATED FV548 STEEL

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ABSTRACT

A nickel, silicon and niobium-rich phase observed in heavy ion and neutron-irradiated FV548 steel has been identified as G-phase. It is suggested that the precipitation of nickel and silicon rich phases in austenitic steels during irradiation could reduce the efficiency of silicon as a void swelling inhibitor and may even have a deleterious effect on the long term swelling behaviour of high silicon casts.

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TABLE

Table 1 Chemical Analyses (wt.%) of FV548 Casts

ILLUSTRATIONS

- Figure 1 Voids and precipitates in VEC-irradiated FV548 steel. Precipitates at (A) showing Moire fringes
- 2 EDX spectrum from a precipitate in irradiated FV548 steel
 - 3 CBDP showing matrix and precipitate $\langle 112 \rangle$ zones. Matrix spots are marked with dots

FV548 is a niobium stabilised austenitic steel which is a candidate material for fast reactor core components (1). The irradiation damage structures and the void swelling produced in FV548 steel irradiated with fast neutrons, electrons and heavy ions have been studied in some detail. Much of the void simulation programme on FV548 steel (2,3,4,5) has been concentrated on a single cast, (VA447, see Table 1), although a number of other casts have also been studied more recently.

It is generally found that a feature of the structure of neutron and ion irradiated FV548 is a very fine dispersion of intragranular niobium carbide (NbC) precipitates which appear to have a marked effect on the void swelling behaviour of the steel (4). NbC is generally the only intragranular precipitate formed in ion-irradiated specimens. However, one of the casts (VA3639, see Table 1) recently irradiated to 60 dpa at 600°C using 46.5 MeV Ni^{6+} ions in the Harwell Variable Energy Cyclotron (VEC) contained, in addition to fine NbC particles, relatively coarse intragranular precipitates which could not be identified as NbC. The precipitates were observed in both 1150°C solution treated and in 20% cold worked specimens of the cast after irradiation.

Transmission electron microscopy showed that Moiré fringes normal to the operating g-vector could be produced in some, but not all of the coarse precipitates (Fig. 1). Such behaviour indicates that these precipitates had a parallel orientation relationship with the austenite matrix and lattice spacings only marginally different from those of the austenite. However, conventional selected area diffraction yielded only limited structural information on the precipitates.

Specimens containing the precipitates were subsequently examined in a Philips EM400 electron microscope fitted with a probe-forming objective lens and an energy dispersive X-ray analyser (EDX).

Figure 2 shows an EDX spectrum obtained from a precipitate. Although the spectrum contains contributions from iron, chromium and manganese in the austenite matrix, it is clear that the precipitates were rich in nickel, niobium and silicon.

Convergent beam diffraction patterns (CBDP) were obtained from several precipitate particles and from the austenite matrix. All the CBDPs obtained from the precipitates were consistent with a face-centred cubic structure. Figure 3 shows a CBDP illustrating parallel precipitate and austenite $\langle 112 \rangle$ zones, from which it can be seen that the precipitate lattice parameter is slightly greater than three times that of the matrix. Using matrix CBDPs for comparison, and assuming an austenite lattice parameter of 0.36 nm, the precipitate lattice parameter was calculated to be 1.115 ± 0.010 nm.

There are three known precipitate phases which have structures which may be interpreted as face-centred cubic and lattice parameters of the right order. The M_{23}C_6 carbide is face-centred cubic, has a lattice parameter of ~ 1.066 nm, and is generally iron, chromium or molybdenum rich. A lattice parameter of 1.115 nm and a high nickel content is therefore not consistent with M_{23}C_6 . The M_6C carbide has a lattice parameter of ~ 1.108 nm and a diamond cubic structure. Double diffraction effects can lead to

confusion between diffraction patterns obtained from the diamond cubic and face-centred cubic structures. However, we found no missing or weak spots in CBDPs obtained from the precipitates described here; the CBDPs were always entirely consistent with an f.c.c. structure. Furthermore the high nickel content of the precipitates is also inconsistent with M_6C . The third possible phase is a silicide of the type generally referred to as G-phase. A number of G-phases are known to exist, and they include $Nb_6Ni_{16}Si_7$ which is face-centred cubic and has a reported lattice parameter of 1.125 nm (6). Clearly the structure, lattice parameter and composition of the precipitate observed in irradiated FV548 indicate that of the three known possibilities, the G-phase is the only one which is consistent with all our observations.

Our observation of G-phase in FV548 steel after heavy ion irradiation has led us to carefully examine unstressed specimens of the steel irradiated with neutrons in the Dounreay Fast Reactor (DFR). EDX and convergent beam diffraction clearly show intragranular G-phase precipitation, frequently but not always associated with voids in specimens from two casts of FV548 (VA447 and 01388) after irradiation to 16 dpa or 30 dpa at temperatures up to $\sim 560^\circ\text{C}$. Furthermore, grain boundaries in these specimens also contain copious amounts of G-phase.

One of the two DFR-irradiated casts in which G-phase has been observed (cast VA447) is the one examined most extensively in the VEC simulation programme. The absence of noticeable amounts of G-phase in heavy ion irradiated specimens from this cast and its presence in equivalent specimens irradiated with neutrons suggests that G-phase is more likely to form under neutron irradiation than during irradiation with heavy ions.

It is becoming increasingly clear that nickel and silicon rich phases form in a number of austenitic steels and nickel-base alloys during irradiation. The nature of the nickel and silicon rich phase formed varies with alloy composition. In Type 316 steel Ni_3Si is formed (7), while the present work shows that in a niobium stabilised steel such as FV548, one of the phases formed is G-phase. Such precipitation is almost certainly enhanced by the segregation of silicon (and possibly nickel) to point defect sinks during irradiation. (For a recent review, see (8)).

Silicon is widely regarded (8,10,11) as a suppressor of void swelling in austenitic alloys, probably exerting its influence by a point defect trapping mechanism. The precipitation of nickel and silicon-rich phases during irradiation and the consequent reduction in the soluble silicon level raises doubts about the long term efficacy of silicon as a swelling inhibitor.

Furthermore, since the phases formed are frequently Ni-rich with respect to silicon (Ni_3Si , $Nb_6Ni_{16}Si_{17}$) there is a possibility that sufficient nickel may be removed from the matrix to affect the stability of the austenite in which the Ni and Si rich phases form. This may be particularly important in alloys such as Type 316 and FV548 whose composition is such that the austenite already has a tendency to instability. (Typical silicon contents of ~ 0.5 wt.% could, if all the silicon formed Ni_3Si or G-phase,

remove ~ 3 wt.% or ~ 2 wt.% Ni, respectively from the austenite matrix).

It has been suggested (12) that the irradiation-induced void swelling in austenitic alloys may be enhanced in two-phase systems. There is therefore a further possibility that by removing nickel from solution and thereby reducing the stability of the austenite phase, silicon may in fact have long term deleterious effects on void swelling. This may be particularly true in alloys with increased silicon contents which have been developed for void swelling resistance.

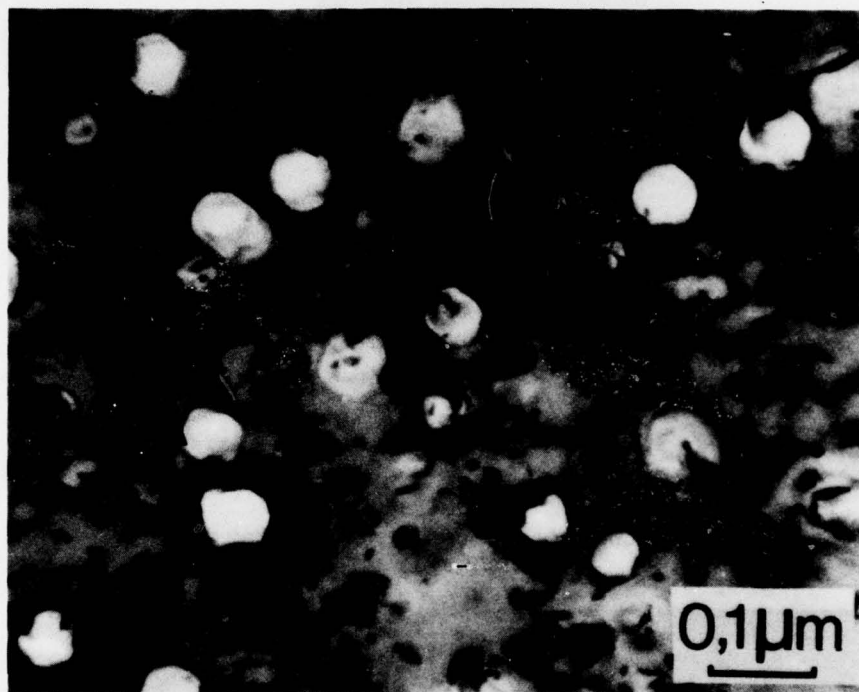
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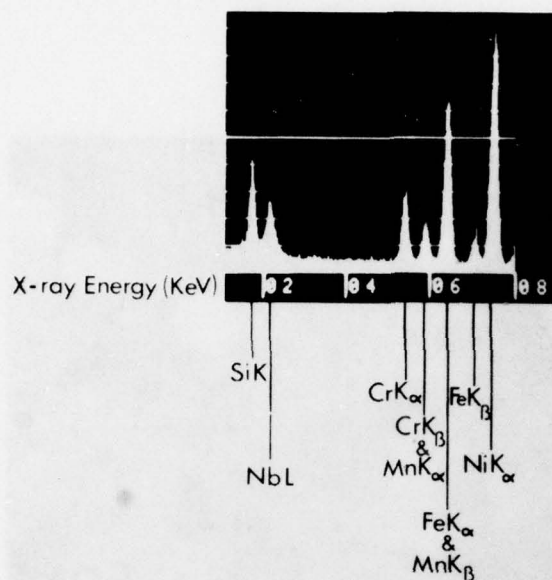
TABLE 1

Chemical Analyses (wt.%) of FV548 Casts

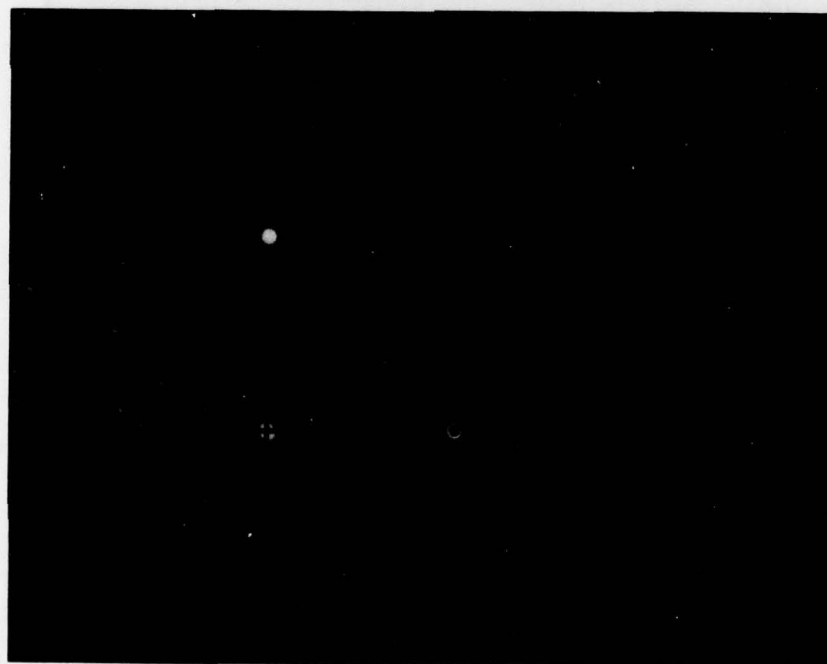
Element Cast No.	C	Mn	P	S	Si	Ni	Cr	Mo	Nb	B	N
VA447	0.11	1.14	0.009	0.006	0.35	11.8	16.5	1.44	0.92	0.0025	0.024
VA3639	0.11	1.15	0.009	0.011	0.36	11.6	17.0	1.4	1.09	0.002	0.019
01388	0.08	1.06	0.017	0.008	0.45	11.5	16.26	1.40	1.02		



AERE - M 3006 Fig. 1
Voids and precipitates in VEC-irradiated FV548 steel. Precipitates at (A)
showing Moiré fringes



AERE - M 3006 Fig. 2
EDX spectrum from a precipitate in irradiated
FV548 steel



AERE - M 3006 Fig. 3
CBDP showing matrix and precipitate $\langle 112 \rangle$ zones. Matrix spots are marked
with dots